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**AFRPL-TR-69-82**

# **LONG-TERM STORABILITY OF PROPELLANT TANKAGE AND COMPONENTS**

**J. E. BRANIGAN**

**AD857651**

## **TECHNICAL REPORT AFRPL-TR-69-82**

**APRIL 1969**

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AFRPL-TR-69-82

LONG-TERM STORABILITY OF PROPELLANT TANKAGE  
AND COMPONENTS

John E. Branigan

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## FOREWORD

This report covers the testing of liquid rocket propellant tankage and propulsion subsystems to evaluate their long-term storage characteristics. The testing is being conducted by the Air Force Rocket Propulsion Laboratory, Edwards, California, under project number 305805FRJ. The testing is being conducted in test area 1-40. The project engineer is Lt Richard B. Mears, and the time period covered by this report is from January 1967 through March 1969.

This report has been reviewed and approved.

EDWARD E. STEIN, Chief  
Propulsion Subsystems Branch  
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## ABSTRACT

Air Force weapons systems require long-term maintenance-free storage, preferably under uncontrolled environmental conditions. Liquid propulsion system components must be capable of satisfactory operation after years of exposure to highly reactive propellants while retaining the propellant without leakage under severe ambient conditions of temperature and relative humidity. Oxidizer leakage caused by improper component design and severe ambient storage conditions has presented serious operational problems.

The Air Force Rocket Propulsion Laboratory (AFRPL) has initiated a program to investigate the storability of liquid system components and tankage under extreme conditions of relative humidity and temperature. A variety of system components and tankage materials are being evaluated for long-term storability with storable liquid rocket fuels and oxidizers. Storage conditions are 85°F temperature and 85 percent relative humidity for oxidizer systems and 70 to 150°F temperature for fuel systems. The propellants under test are N<sub>2</sub>O<sub>4</sub>, ClF<sub>3</sub>, ClF<sub>5</sub>, and MHF-5. Tankage materials under test are various alloys of aluminum, steel, and titanium.

The results of almost 2 years of testing on a representative number of tankage materials have indicated that leakage of propellant can occur as a result of improper weld joint design, inadequate quality control in fabrication and inadequate acceptance leakage testing. Factors which can contribute to the development of oxidizer leakage are a high ambient relative humidity (>30 percent) and stress corrosion cracking susceptibility of the tank material in combination with the propellant and trace quantities of foreign compounds/elements in the propellant.

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## SECTION I

### INTRODUCTION

Experience with liquid propellant rocket feed systems has shown that leakage of oxidizers can occur and constitute a difficult problem under certain environmental conditions. In propellant tankage, leakage has been observed in or adjacent to weldments, and more specifically in weldments in which a double heat cycle has occurred, either by repeated welding to effect a repair or by intersecting weldments. It has been shown experimentally (1) in the case of  $N_2O_4$ , that when a vapor leak occurs the result is drastically influenced by the relative humidity of the atmosphere surrounding the tanks. If the relative humidity is on the order of 30 percent or lower, the nitric oxide vapor, which is the leaking fluid, dissipates into the atmosphere and does nothing to aggravate the leakage. If the relative humidity is in the order of 40 percent or greater, however, it does not dissipate, but rather hydrolyzes, forming dilute nitric acid on the exterior surface in the immediate vicinity of the leak (Figure 1). The action of the nitric acid is to enlarge the original leakage path, working inward toward the source of the leak. Eventually, a small, or even minute vapor leak can become a large liquid leak, if it is allowed to proceed. Although a similar detailed experimental program has not been performed with the storable fluorinated oxidizers such as  $ClF_3$  and  $ClF_5$ , an analogous process would be expected with hydrogen fluoride as the hydrolysis product.

In the past, the selection of materials for systems applications has been based on conventional fluid compatibility testing to determine discoloration, pitting, weight loss or gain, notch sensitivity and stress corrosion cracking susceptibility as well as potential degrading effects on the propellant. Even after this thorough analysis and selection process, the material or the processing used in the propellant tankage may not function properly for extended periods or may develop leaks during its storage life. The use of

conventional compatibility criteria, while certainly an essential part of the material selection process, has not served to screen out materials or processes which are not suitable for extended storage of liquid propellants when fabricated into system tankage. The major limitation on interpreting long-term storability effects in realistically severe environmental conditions of storage or service life is the inability of conventional compatibility criteria to predict leakage. The possible exception is the identifying of susceptibility to stress corrosion cracking. In liquid propellant tankage, this susceptibility usually leads to catastrophic rupture of a tank rather than leakage, since few, if any, flight-weight tanks have a "leak before burst capability". Small, undetected pin holes or microcracks could be formed by an attack of the propellant on grain boundary precipitates and inclusions, but would not be detected by weight gain or loss calculations and would probably go undetected. The possibility of such defects forming is greater in the limited-weldability materials where there is a tendency for microcracking. The size and methods of producing test specimens used in compatibility work eliminates many of the manufacturing and quality control problems associated with production systems. Smooth, polished samples, welded or unwelded, are not comparable to fabricated tankage material. No. 2014 T-6 aluminum is compatible with nitrogen tetroxide ( $N_2O_4$  MIL-P-265398), however, experience has shown that  $N_2O_4$  leakage can occur with this 2014 T-6 material, usually in the heat-affected weld zone, in a humid environment (>30 percent). Long periods of storage may affect the functional performance and system reliability of prepackaged liquid propulsion systems. There are many areas to consider in providing data to supplement coupon compatibility testing. Storage conditions must be selected that are representative of system operational conditions. Such factors as humidity and temperature play an important role. A detailed propellant analysis before and after testing is required to evaluate the effects of storage on the propellant. The cleanliness level of test articles must be known for reasons of safety, but equally important, to evaluate the processes which were used to effect this level. Materials and chemicals used for cleaning may have an effect on the system life. In the same

manner, manufacturing processes and quality control standards may impose many unforeseen conditions which vary from one manufacturer to another. Throughout the fabrication of a test article (i. e., during welding, x-ray, dye penetrant inspection and test), all data should be available to result in a meaningful post-failure analysis in the event that leakage occurs. Metal preparation prior to welding may make the difference between a satisfactory or unsatisfactory weld with regard to its ability to contain propellant without leakage. Helium leak testing of systems and the technique of leak testing are very important since small leakage which cannot be detected by x-ray or dye penetrant inspection can lead to propellant leakage under adverse environmental conditions. These variables must be known and controlled in a meaningful storability program.

## SECTION II

### PROGRAM STRUCTURE

The Air Force Rocket Propulsion Laboratory (AFRPL) initiated a program entitled "Packaged System Storability" to supplement laboratory compatibility work. This program deals with evaluation and demonstration of long-term (5 to 10 years) storage of tankage, components and integrated propulsion feed systems with present and advanced propellants. Materials under investigation include aluminum, steel, and titanium alloys. Test systems include tankage, integrated systems, consisting of tankage and feed system components, and complete feed systems including tankage, components, expulsion devices, and gas pressurization systems.

The AFRPL program consists of two basic types of testing: propellant tankage and integrated feed systems. The organization of the program is presented in the succeeding paragraphs.

#### A. PHASE I - SMALL CONTAINER TESTING

Small containers of approximately 1 quart capacity manufactured from aluminum and steel alloys are used to evaluate a particular problem to or evaluate promising materials. These test articles are inexpensive and serve as excellent "screening" devices, but cannot duplicate larger manufacturing and production problems. Figure 2 shows the 3- by 6-inch containers in the test chamber, along with the radioisotope liquid-level gage system used to control the propellant loading operation in all phases of the program. Figure 3 shows the 1-quart containers in the Quonset hut. Figure 4 shows the 301 stainless steel cylinders. This phase is subdivided into three parts:

Part 1 - 3- by 6-inch Containers. Forty containers manufactured from 2014 T-6 aluminum, by Douglas, General Dynamics, Martin, and North American are used to evaluate various approaches to manufacturing 2014 T-6 tankage for containment of N<sub>2</sub>O<sub>4</sub>.

Part 2 - Alcoa 1-quart containers. An Alcoa standard container for material compatibility testing is used to evaluate the storability of various standard and experimental aluminum alloys with  $N_2O_4$ ,  $CIF_3$ , and  $CIF_5$ . The aluminum alloys are 2014 T-6, 6061 T-6, 7007 T-6, 2219 T-81, 5456 T-6, 3003 T-6, and X2021 T-6.

Part 3 - Arde Cylinders. Small cylindrical containers, developed by Arde, Inc., as high-pressure  $CO_2$  cylinders of AISI 301, cryogenically stretch formed stainless steel, are used to evaluate the storability of this material in both an aged and unaged condition with  $N_2O_4$  and  $CIF_5$ .

#### B. PHASE II - AFRFL INTEGRATED SYSTEMS

Figure 5 shows these systems located in the Quonset hut. These systems consist of the major components of a propellant feed system which contact propellant during the storage life of the system. Fifteen-gallon tankage is of 2219 T-81 aluminum and AM350 steel. System components consist of pressure switch, explosive valve and burst disk. Fittings are AFRPL mechanical fittings (MIL-F-27417) and TIG welded joints. Since the tankage material and the component materials are of both aluminum and steels, inter-metal transitions are made using both mechanical fittings and solid-state bonded transition joints. Twenty-four systems have been integrated, 12 using aluminum tankage and 12 using steel tankage for storability evaluation with  $N_2O_4$  and  $CIF_5$ .

#### C. PHASE III - FIFTEEN-GALLON TANKS

Figure 6 shows these tanks located in the Quonset hut. This phase constitutes the tankage test phase of the program in which different metals and alloys are evaluated with  $N_2O_4$  and  $CIF_5$ . A total of 24 were procured: 12 each from the Martin Company and General Dynamics/Convair, from several aluminum, steel and titanium alloys. The tankage was manufactured using large-scale tank production methods. Tankage included dome gore welds, cylindrical and longitudinal welds, which are representative of large tankage design. Manufacturing process records, x-rays, inspec-

tion logs and metallurgical samples of welded and unwelded materials were delivered with the tanks to serve as test article documentation.

#### D. PHASE IV - EXISTING TANKS

Figure 7 shows the two 2219 aluminum tanks. Figure 8 shows the three Bullpup "B" missile tanks. Figure 9 shows the 6061 aluminum Agena tank. Existing tankage manufactured on other AFRPL and government contracts is being considered for inclusion into the storability program. Various materials such as 2219 T-81 aluminum and 2014 T-6 aluminum in tankage sizes up to 400 gallons are included in the program. In addition, an Agena 6061 T-6 aluminum tank has been tested for 3 months with  $N_2O_4$  to simulate the launch site hold requirement of the improved Agena stage. The NASA specification grade  $N_2O_4$  (MSC-PPD-2A) characterized by an NO content between 0.4 and 0.8% was used.

#### E. PHASE V - PREPACKAGE FEED SYSTEM

Figure 10 shows these systems located in the Quonset hut. General Dynamics/Convair developed prepackaged feed systems. These systems include electron-beam-welded 2219 T-81 aluminum tankage with both rolling diaphragm and surface tension screen expulsion devices and liquid propellant gas generator (LPGG), solid propellant gas generator (SPGG) and high-pressure stored gas device (GD) pressurization systems. Systems were loaded with  $N_2O_4$ ,  $CIF_5$ , and MHF-5, delivered to the AFRPL, stored, vibrated at time intervals and finally operated to evaluated system component functioning. After expulsion, the system will undergo destructive analysis in the laboratory.

### SECTION III

#### TEST FACILITY

Storage testing of the N<sub>2</sub>O<sub>4</sub> tanks is conducted in a metal Quonset hut storage test building equipped to provide a constant controlled environment of 85% temperature and 85% relative humidity. The storage test building is insulated by a spray-in-place foam (polyurethane). Environmental conditions are maintained by two evaporative coolers and immersion water heaters. Variations in test conditions are approximately  $\pm 5^{\circ}\text{F}$  temperature and  $\pm 5\%$  relative humidity. Safety provisions in the storage test building consist of a Firex-type water deluge system, large water drain piping, fire detectors, and closed-circuit television monitoring.

## SECTION IV

### PROCEDURES

#### A. TEST ARTICLE INSPECTION

The primary responsibility for quality control and quality assurance of the test articles was vested in the manufacturers of the test articles. To insure high-quality test articles for use in the storability program, as well as to provide a means to conduct a comprehensive, meaningful failure analysis and failure cause determination where failures did occur in testing, procedure specifications governing all aspects of test article manufacture, inspection, and cleaning were either generated or were identified for use in the procurement of all test articles used on the program.

All test articles and test systems, with the exception of the Phase V, Prepackaged Feed Systems, were leak tested and recleaned at the AFRPL to insure against the development of leaks and the introduction of contamination during shipment of the test articles from the manufacturer. The procedures followed at the AFRPL for these processes were similar to the procedures used by the manufacturers of the test articles.

#### B. PROPELLANT LOADING

The following procedures were used to load propellant into the test tanks and systems, with the exception of the Phase V, Prepackaged Feed Systems, which were loaded with propellant by the manufacturer prior to delivery.

$\text{N}_2\text{O}_4$  - The  $\text{N}_2\text{O}_4$  tanks and systems were loaded by individually opening a vent valve on the top of the test system and a fill valve on the bottom. The liquid level was controlled by means of a radioisotope ( $\text{Co}^{60}$ ) liquid-level sensor system attached to the tank at the desired level. This method of level control proved to be entirely satisfactory.

CIF<sub>3</sub> - The procedure for loading CIF<sub>3</sub> was identical to that used for N<sub>2</sub>O<sub>4</sub>.

CIF<sub>5</sub> - The procedure used to load N<sub>2</sub>O<sub>4</sub> and CIF<sub>3</sub> was found to be unsuitable for loading CIF<sub>5</sub> because it resulted in large CIF<sub>5</sub> losses. This was due to the high vapor pressure of CIF<sub>5</sub>. The procedure used consisted of cooling the tank with low-temperature gaseous nitrogen and pressure-transferring the CIF<sub>5</sub> into the test tank with the valve closed. In addition to eliminating the loss of CIF<sub>5</sub> this procedure significantly reduced the time required to load.

#### C. TEST ARTICLE PASSIVATION

All interhalogen tanks and systems were passivated with gaseous fluorine prior to loading with propellant. The procedure used consisted of incrementally increasing the partial pressure (mole fraction) from 25% to 100% in 25% increments, with a hold period of 4 hours at each increment.

## SECTION V

### DISCUSSION OF RESULTS

A summary of all test articles and the results to date is presented in Tables I through V.

#### A. PHASE I

The 3-by 6-inch system has had five failures. The first four leaks were observed after storage at 85°F and 85% relative humidity for 5 days. These containers were helium leak-checked after failure under the same conditions as before leakage occurred, using 90 to 100% mixture and nitrogen and helium and hand probe. High leakage rates were detected. Preliminary analysis at the AFRPL determined that there was evidence of microcracks in the heat-affected zone near the failure area. These three containers were sent to the Air Force Materials Laboratory (AFML) for destructive testing and failure analysis. The AFML reported that the failures were a result of poor container end-plate joint design which resulted in lack of penetration in the flat 1/4-inch plate to the .064-inch-thick cylindrical section. The AFML report is presented as Appendix I to this report. This design was characteristic of containers manufactured by two contractors. The designs by two other contractors eliminated the problem by cutting the 1/4-inch end plate to .064-inch at the weld area. To date, there has been only one failure in this area on the improved design containers. No analysis has been accomplished on this tank.

#### B. PHASE II

These systems have been in test with  $N_2O_4$  since 9 May 1968. As of this date, no leakage has occurred in either the tanks or in any of the system fittings or components.

#### C. PHASE III

There has been a total of nine propellant leaks with  $N_2O_4$  and five with  $CIF_5$ . There are six tanks remaining in  $N_2O_4$  test; in  $CIF_5$ , none are

actually in test, although three are ready to be tested. Of four 2014 T-6 aluminum tanks, one leaked within 1 month and the remaining three are still in test. An analysis of the one leak revealed that excessive chemical attack had occurred in the region of the larger of two leaks on the outside of the tank. This attack occurred primarily along the weld heat-affected zone, after the tank had leaked.  $\text{NO}_2$  reacted with the atmospheric moisture, forming dilute nitric acid which rapidly attacked the aluminum alloy. The major leak occurred at a "tee" weld intersection. Numerous gas voids with an interdendritic crack network running from hole to hole were found. The weld played a major role in the failure initially. A sound weld, of good quality, would not be expected to suffer corrosion in  $\text{N}_2\text{O}_4$ . An intergranular corrosion coupled with the presence of a stress field and some contamination, probably surface contamination, was believed to be the cause of the failure. The reports of the failure analyses performed to date are presented as Appendixes I and II to this report. The same conclusions were presented in a third failure analysis, which is reported in Reference 2. Reports of the failure analyses of the remaining leaking tanks will be published as they become available.

All the titanium tanks leaked in less than 35 days after loading with  $\text{N}_2\text{O}_4$ . Both the 6Al-4V and the 5Al-2.5 titanium alloys in the annealed condition were tested. Consideration was given to the use of the NASA grade  $\text{N}_2\text{O}_4$  (MSC-PPC-2A specification (green)) in the loading of the titanium tanks because of the stress corrosion problem encountered with MIL-P-26539B specification  $\text{N}_2\text{O}_4$  (brown). The stress levels in the tankage, based on nominal loads and thickness, however, were considerably below the threshold for stress corrosion cracking reported (16Ksi versus 90Ksi). The test temperature was to be significantly below the temperatures at which problems were encountered ( $85^\circ\text{F}$  versus  $110^\circ\text{F}$ ). On the basis of these two considerations, stress corrosion was not thought to be significant and the tanks were loaded with MIL-P-26539B specification  $\text{N}_2\text{O}_4$  (brown). Fracture analysis of two of the five leaks revealed that stress corrosion cracking was present in the failure area and was very

probably responsible for the leaks. The reports on these analyses are presented as Appendix II of this report. It is quite likely that warping loads which were introduced by welding in the leak area resulted in residual stresses which significantly increased the local stress level above the calculated general membrane stress level. A repair had been made of the weld in the vicinity of the leak and some distortion was visually evident. A failure analysis has been initiated to do similar analyses on the remaining tanks which have leaked. The analyses that will be performed are as follows. An initial micro-examination will be made to observe gross effects with regard to corrosion appearance and leakage site appearance. The corrosion products will be analyzed and a helium leak test performed to pinpoint the location of the leak. The leak will then be radiographed and sectioned to expose the fracture surface. Subsequent analyses will depend on findings to this point. Optical photomicrographs will be taken as a minimum. If further analysis is warranted, electron fractrography and electron microprobe work will be done.

#### D. PHASE IV

Two types of tanks are currently in test with MSC-PPD-2A specification  $N_2O_4$  (green). The first type is an aluminum, bipropellant tank, approximately 44 inches in diameter and 67 inches in length. The tank is made of 2219 T-81 aluminum and includes a common dome between the fuel and oxidizer. For storability testing,  $N_2O_4$  (MSC-PPD-2A specification (green)) is loaded in both the fuel and oxidizer portions of the tank. Two of these have been in test since 21 May 1969 with no leakage to date.

The second type of tankage is a standard Bullpup "B" missile with all ordnance devices omitted.  $N_2O_4$  (MSC-PPD-2A specification (green)) is loaded in both the oxidizer portion (normally IRFNA) and the fuel portions of the tank (normally MAF-2). The tank material is 2014 T-6 aluminum. Three Bullpup "B" missile tanks have been in test since 21 May 1968 with no leakage to date.

A short-duration storability test of a standard Agena tank was conducted with MSC-PPD-2A specification (green)  $N_2O_4$  (Figure 7). Oxidizer was loaded in both the oxidizer portion of the tank (normally IRFNA) and the fuel portion (normally UDMH). The Agena tankage is 6061 T-6 aluminum with a common dome between the fuel and oxidizer. Testing was conducted for approximately 90 days at 85% relative humidity and 85°F temperature with no leakage occurring.

#### E. PHASE V

Two prepackaged systems suffered leakage during storage testing in identical modes of failure: one oxidizer ( $CIF_5$ ) and one fuel (MHF-5). A small (1/4-inch-diameter tube approximately 2 inches long welded to the bottom of the tank serves as a propellant loading tube. The procedure used by General Dynamics/Convair was to install "vee" shaped clamps to the tube after the propellant was loaded and weld a plug into the end of the tube. Leakage occurred at this plug weld. The oxidizer system was destroyed because of the hazard of attempting a repair with  $CIF_5$ . The fuel system was returned to test after it was repaired by "repinchng" the tube and replacing the welded plug with an AN fitting.

The oxidizer systems are located in the modified Quonset hut and the test conditions are 85% relative humidity and 85°F temperature. The fuel systems are located in a temperature-controlled building and are cycled between ambient temperature (70°F) and 150°F.

## SECTION VI

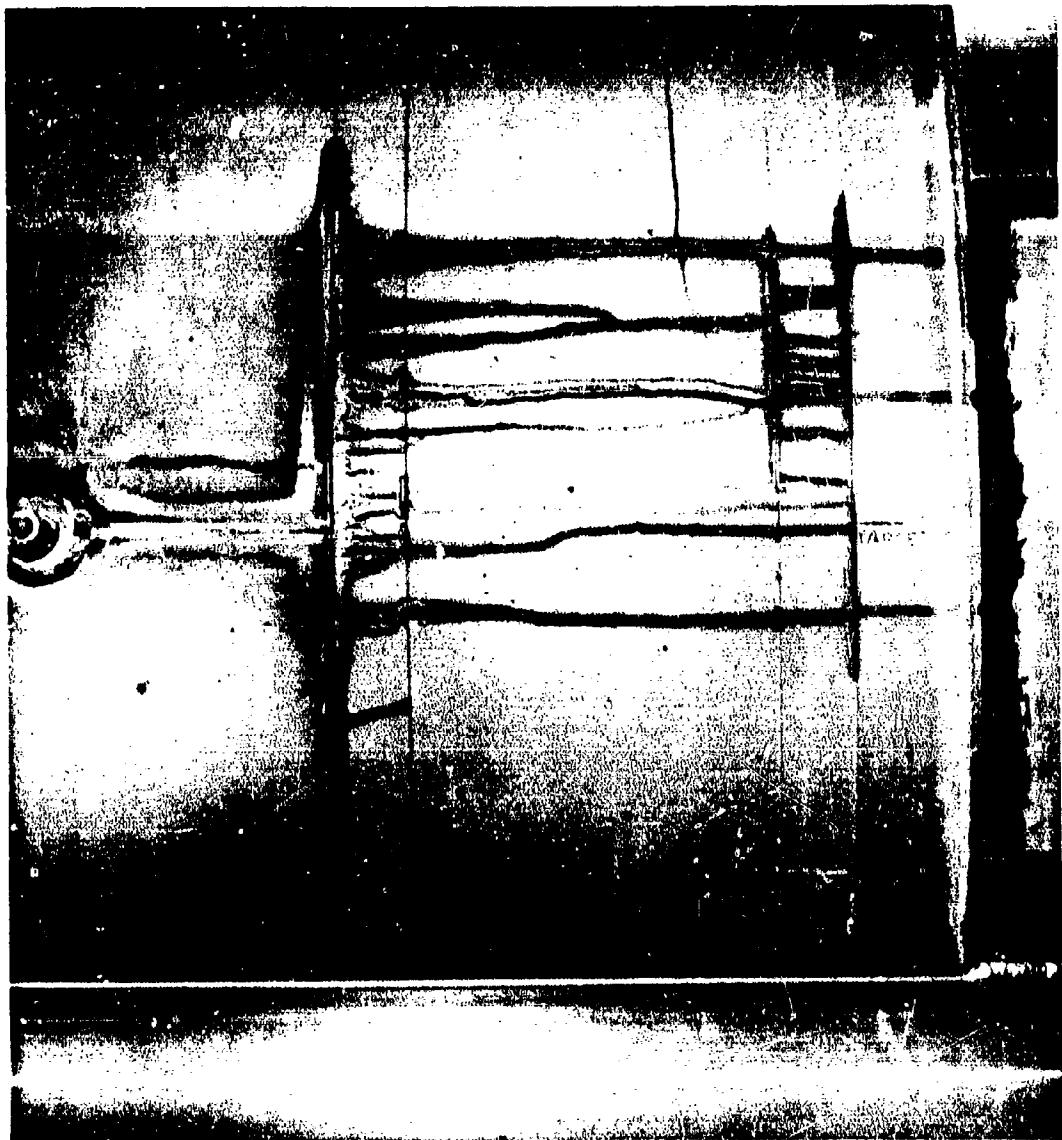
### SUMMARY

A representative number of liquid propellant tankage metals, fabricated by typical production methods of forming and welding and subjected to the same degree of inspection and quality control documentation as production tankage, have been evaluated in storage testing for nearly 2 years. A number of leaks have occurred in all three types of tankage metals—aluminum, steel, and titanium. However, a significant number of tanks have successfully held propellant for the same time period in both  $N_2O_4$  and  $CIF_5$ . In general, the failures have been attributed more to quality of fabrication, welding in particular, than to the metals themselves. It is realized that those metals which are more difficult to weld are undoubtedly more susceptible to poor quality welds than metals which are more easily welded. Weld repairs are very susceptible to leakage, and although repairs may be necessary with any tankage metal, difficult-to-weld metals are more likely to require repair.

The titanium tankage leaks, being caused by stress corrosion, were related to the grade of propellant used in the test. This testing is being repeated using a grade of propellant which should inhibit stress corrosion cracking and thereby prevent leakage.

Solid-state bonded tanks offer great promise by the elimination of the single major cause of propellant tankage leaks, the cast structure of a fusion weld. A solid-state bonded joint is metallurgically identical to the parent metal of which the joint is made. The microcracking, porosity, and other imperfections which occur in fusion welds are eliminated from a good solid-state bonded joint. Since leakage is intimately associated with weld quality, improvements in joints technology will produce corresponding improvements in eliminating leakage.

Figure 1. Hydroscopic Action of  $\text{NO}_2$  Vapor



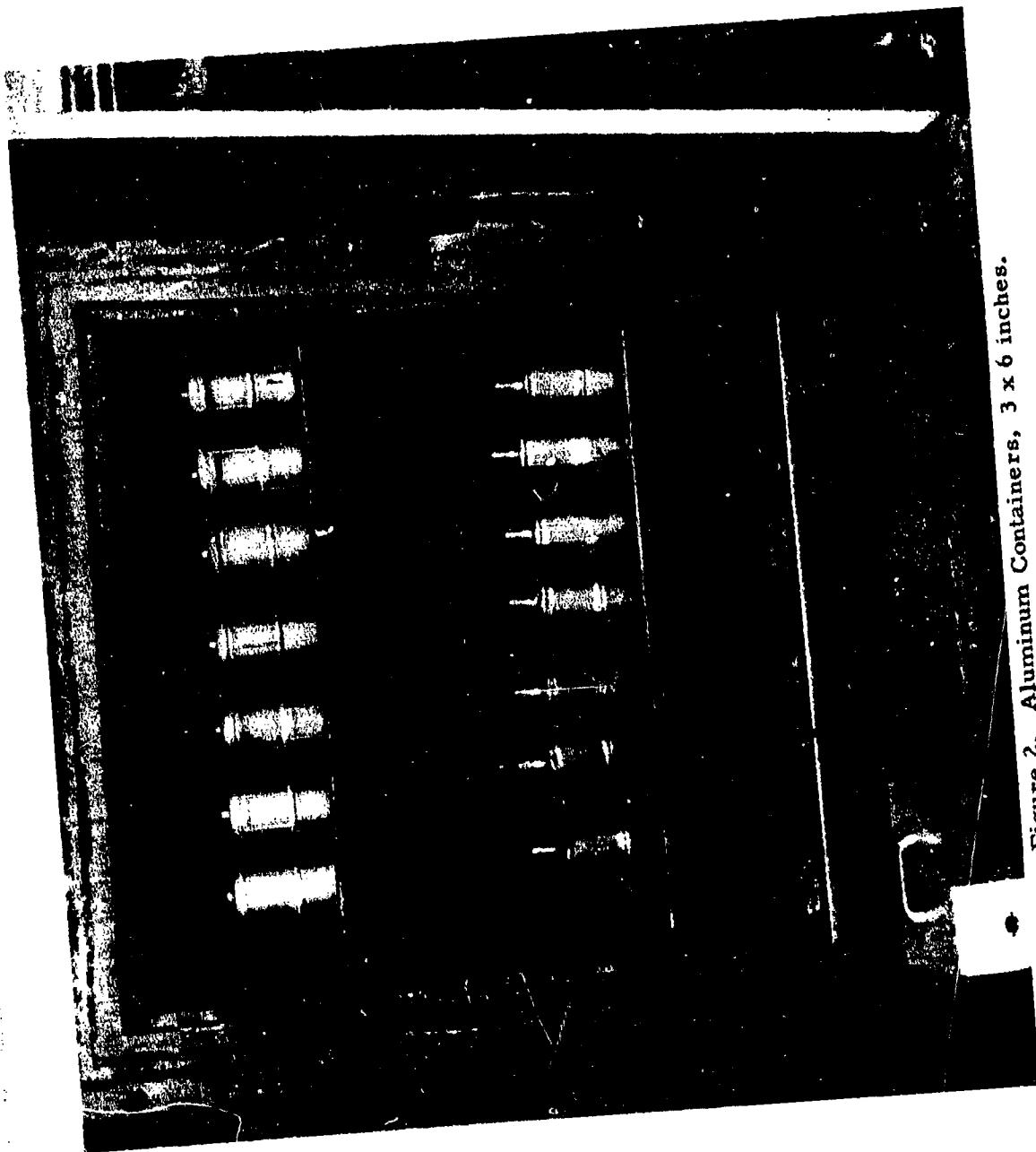


Figure 2. Aluminum Containers, 3 x 6 inches.

Figure 3. Alcoa 1-Quart Containers



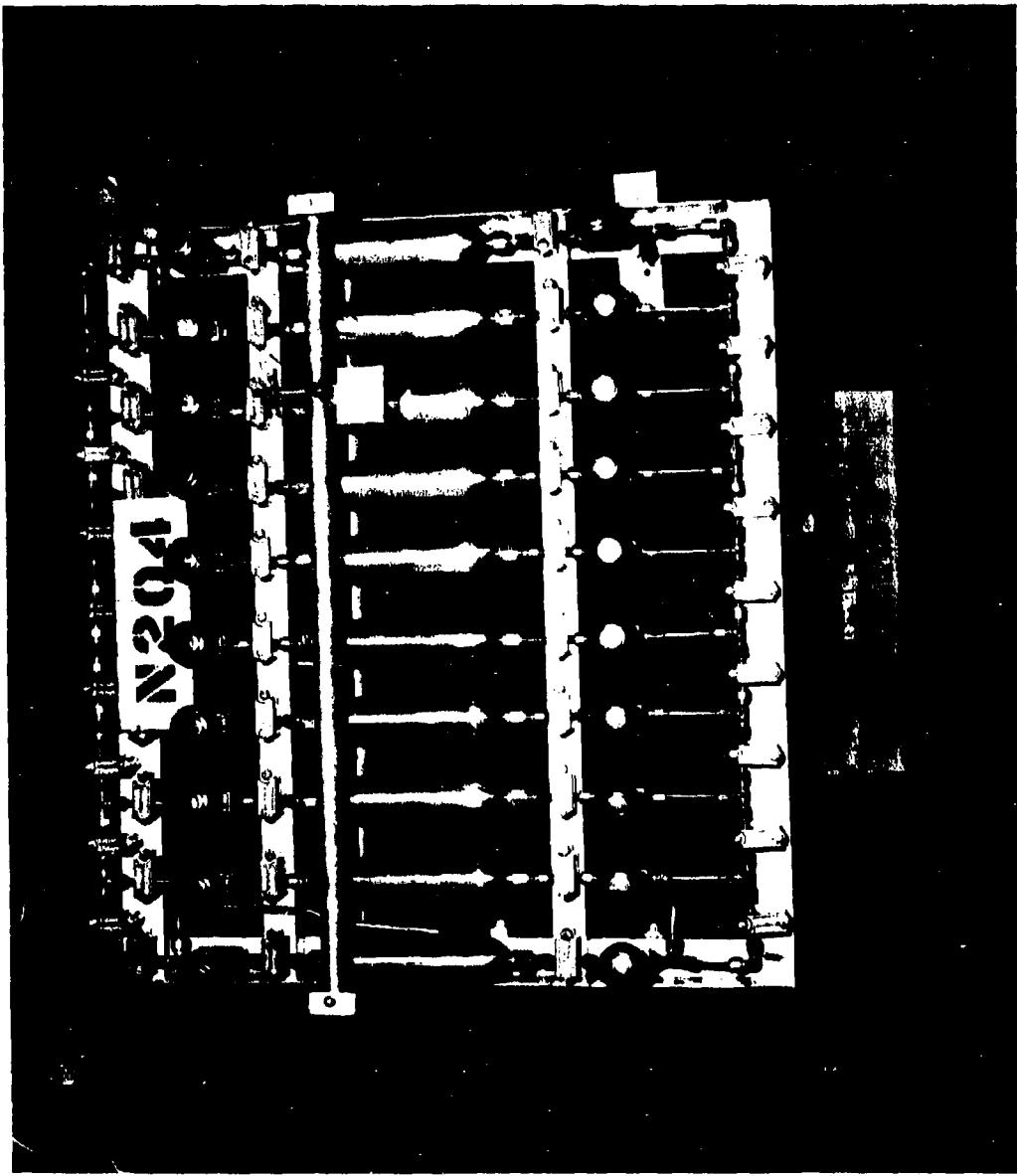
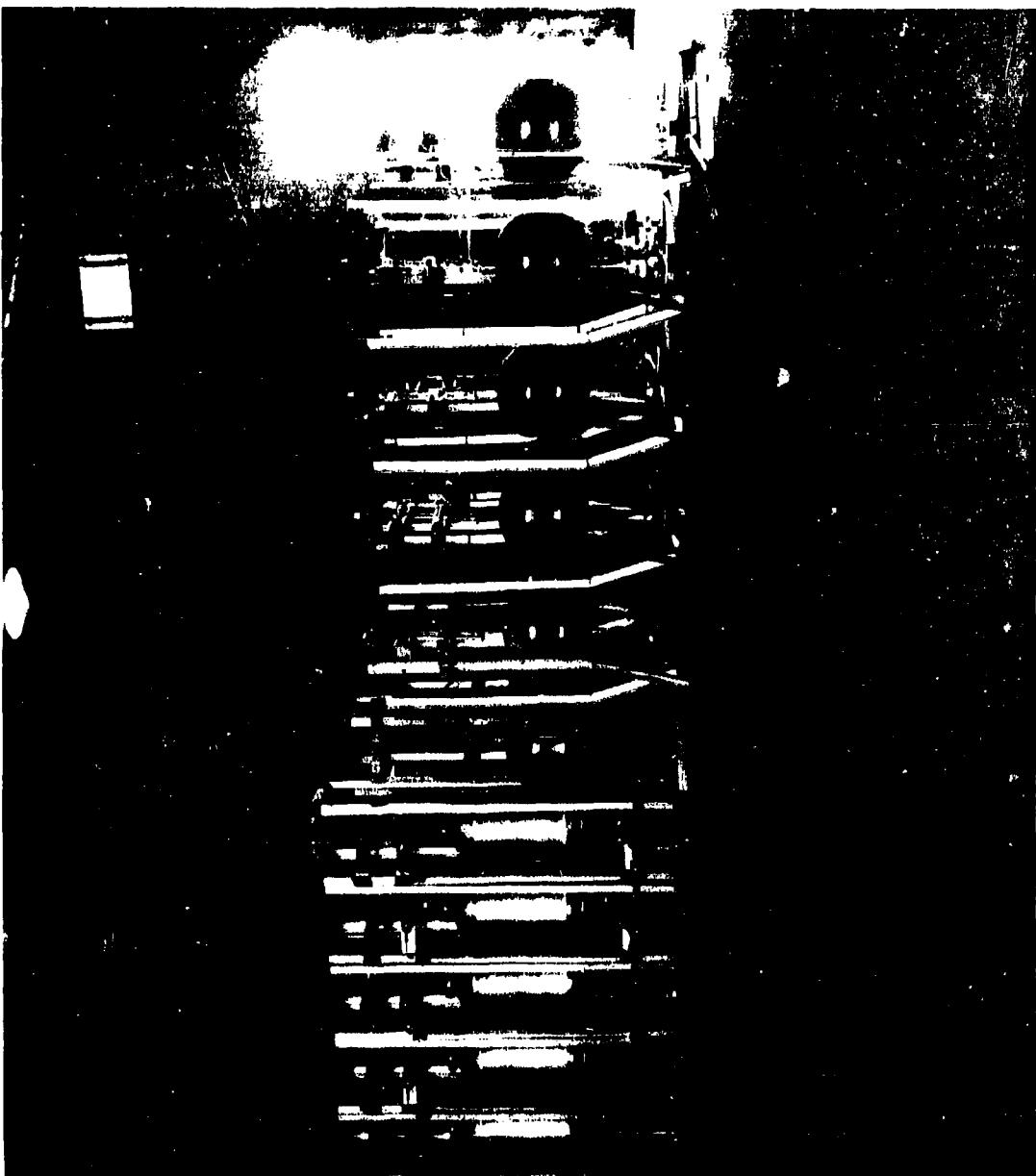


Figure 4. Arde 301 Stainless Steel Cylinders

**Figure 5. AFRPL Integrated Systems**



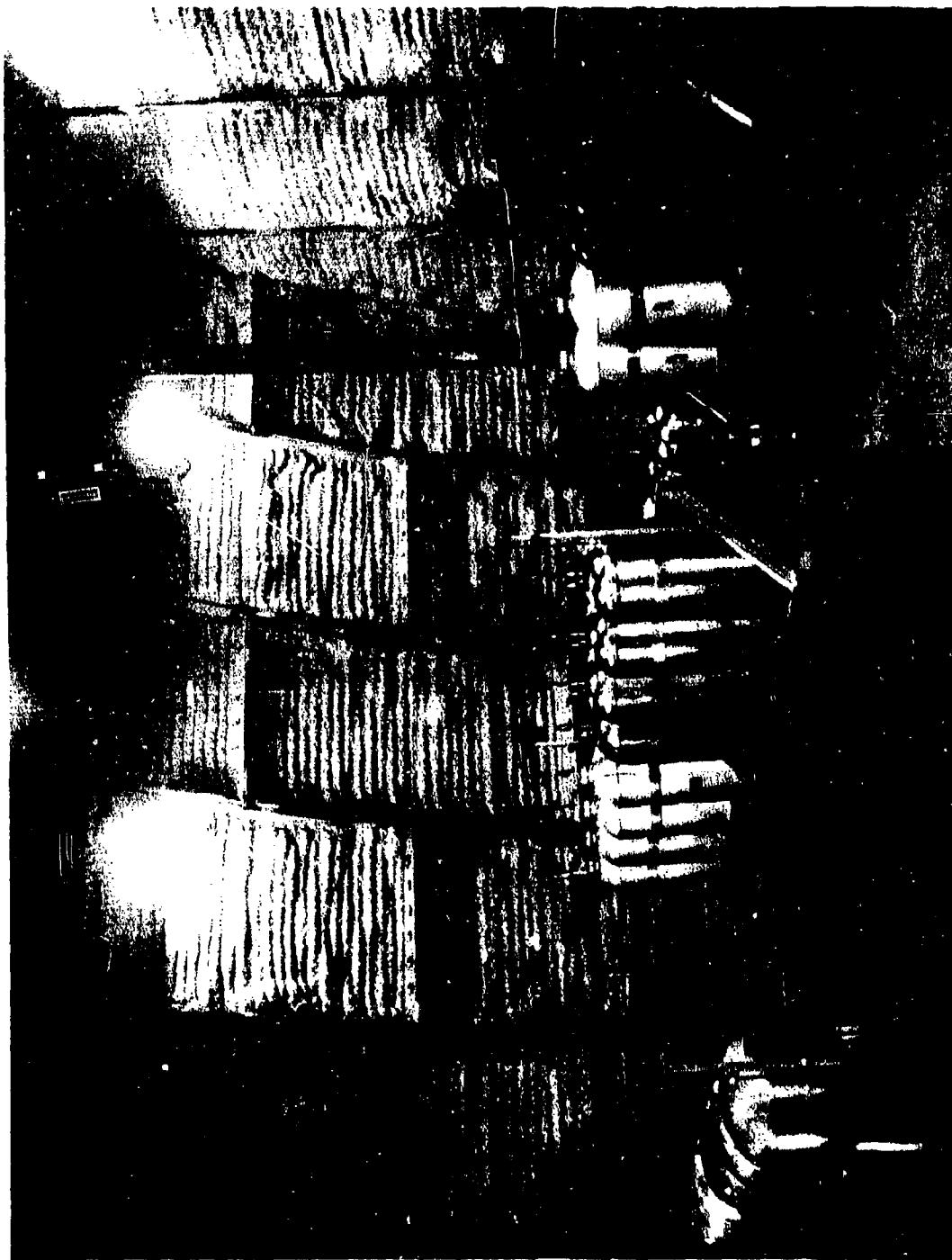
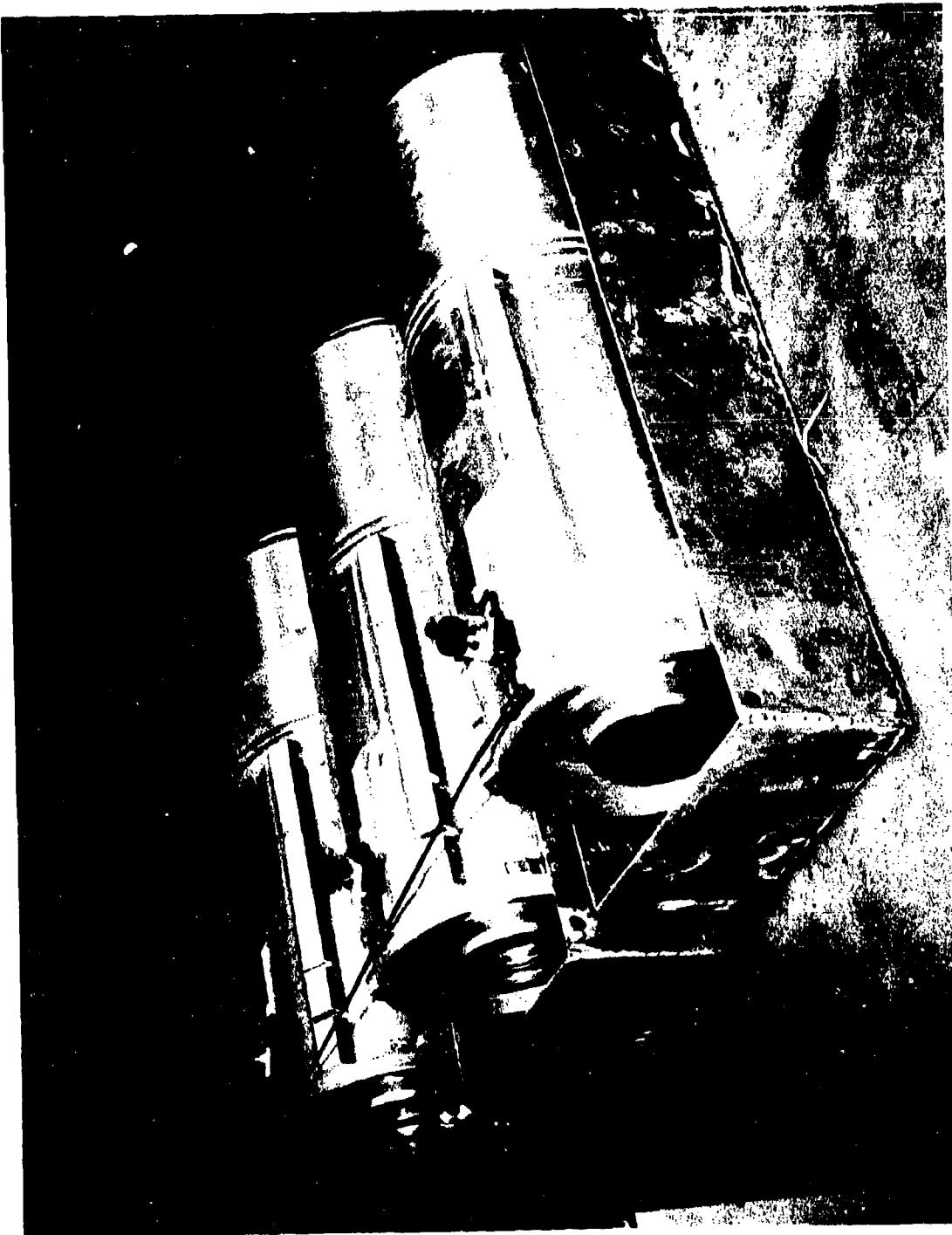


Figure 6. Fifteen-Gallon Tanks



Figure 7. No 2219 Aluminum Tanks

Figure 8. Bullpup "B" Missile Tanks



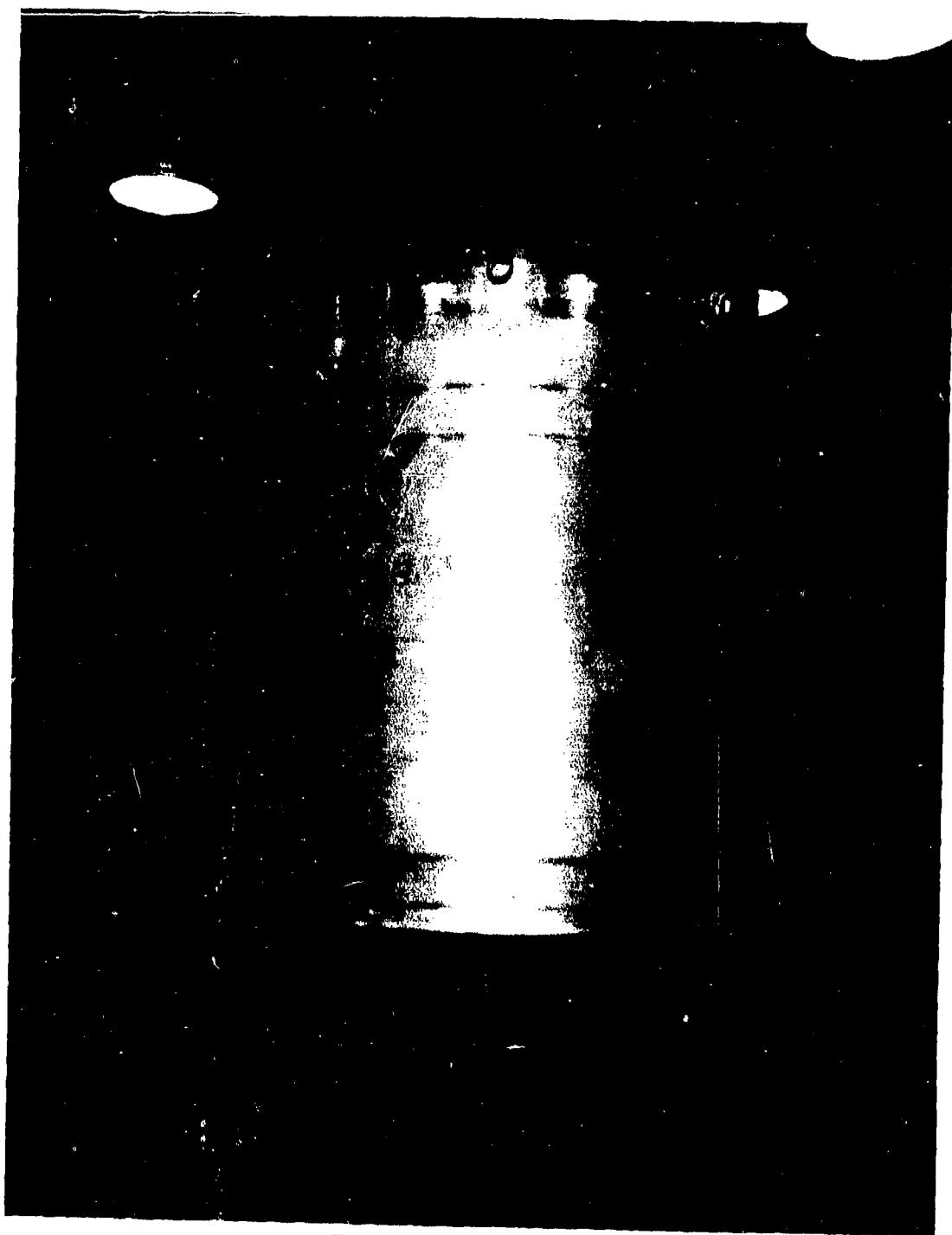


Figure 9. Agena Tank

Figure 10. Prepackaged Feed Systems

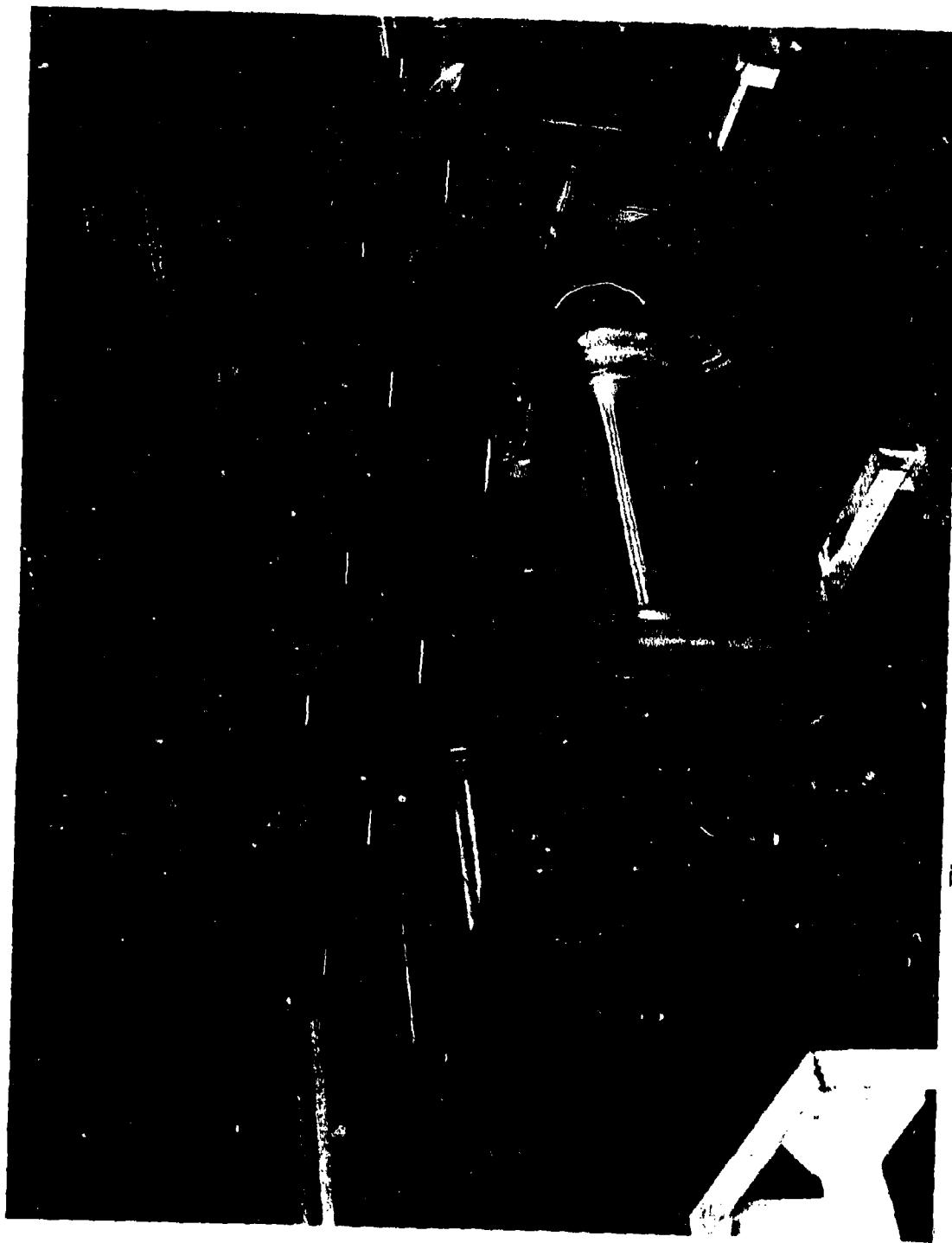


TABLE I. PHASE I - SUMMARY OF RESULTS

Propellant	Tank	Quantity	Tank Material	Test Initiated	Test Terminated	Days In Test
N <sub>2</sub> O <sub>4</sub>	3" X 6"	4	2014-T6	9- 7-66	9-12-66	5
N <sub>2</sub> O <sub>4</sub>	3" X 6"	1	2014-T6	1- 3-67	1- 5-67	2
N <sub>2</sub> O <sub>4</sub>	3" X 6"	23	2014-T6	1- 3-67	In Test	
N <sub>2</sub> O <sub>4</sub>	Alcoa 1-qt	6	2014-T6	12- 5-66	In Test	
N <sub>2</sub> O <sub>4</sub>	Alcoa 1-qt	3	6061-T6	12- 5-66	In Test	
N <sub>2</sub> O <sub>4</sub>	Alcoa 1-qt	2	2219-T6	12- 5-66	In Test	
N <sub>2</sub> O <sub>4</sub>	Alcoa 1-qt	1	7007-T6	12- 5-66	In Test	
N <sub>2</sub> O <sub>4</sub>	Alcoa 1-qt	2	2021-T6	12- 5-66	In Test	
N <sub>2</sub> O <sub>4</sub>	Alcoa 1-qt	2	5456-T6	12- 5-66	In Test	
N <sub>2</sub> O <sub>4</sub>	Arde 1-pt Cryo Form	5	AISI 301 Aged	6-21-67	In Test	
N <sub>2</sub> O <sub>4</sub>	Arde 1-pt Cryo Form	5	AISI 301 Unaged	6-21-67	In Test	
C1F <sub>5</sub>	Alcoa 1-qt	1	6061-T6	3-14-67	7-16-68	485
C1F <sub>5</sub>	Alcoa 1-qt	1	6061-T6	3-14-67	In Test	
C1F <sub>5</sub>	Alcoa 1-qt	7	2014-T6	3-14-67	In Test	
C1F <sub>5</sub>	Alcoa 1-qt	2	2219-T6	3-14-67	In Test	
C1F <sub>5</sub>	Alcoa 1-qt	1	3003-T6	3-14-67	In Test	

TABLE II. PHASE II - SUMMARY OF RESULTS

Propellant	Tank	Quantity	Material	Components	Fittings	Test Initiated	Test Terminated	Days in Test
N <sub>2</sub> O <sub>4</sub>	Martin 15 Ga.	1	2219	Burst Disk	AFRPL	5-9-68		In Test
N <sub>2</sub> O <sub>4</sub>	Martin 15 Gal	5	2219	PR.SW.EX.VAL	Welded Trans	5-9-68		In Test
N <sub>2</sub> O <sub>4</sub>	Convair 15 Gal	1	AM350	Burst Disk	AFRPL	5-9-68		In Test
N <sub>2</sub> O <sub>4</sub>	Convair 15 Gal	5	AM350	PR.SW.EX.VAL	Welded Trans	5-9-68		In Test
CIF <sub>5</sub>	Martin 15 Gal	1	2219	Burst Disk	AFRPL	11-12-68	11-13-68	1 Day
CIF <sub>5</sub>	Martin 15 Gal	1	2219	PR.SW.EX.VAL	Welded Trans	11-13-68	11-15-68	2 Days
CIF <sub>5</sub>	Martin 15 Gal	4	2219	Burst Disk	PR.SW.EX.VAL	11-13-68	11-18-68	5 Days
CIF <sub>5</sub>	Convair 15 Gal	1	AM350	Burst Disk	AFRPL	11-13-68		In Test
CIF <sub>5</sub>	Convair 15 Gal	2	AM350	PR.SW.EX.VAL	Welded Trans	11-14-68	11-18-68	4 Days
CIF <sub>5</sub>	Convair 15 Gal	2	AM350	PR.SW.EX.VAL	Welded Trans	11-13-68		In Test
CIF <sub>5</sub>	Convair 15 Gal	1	AM350	PR.SW.EX.VAL	Welded Trans	11-14-68		In Test

PR.SW. Pressure Switch  
 EX.VAL Explosive Valve  
 Trans. Transition Joint

TABLE III. PHASE III - SUMMARY OF RESULTS

Propellant	Tank	Quantity	Material	Test Initiated	Test Terminated	Days
N <sub>2</sub> O <sub>4</sub>	Martin	1	2014-T6	1-3-67	1-25-67	22
N <sub>2</sub> O <sub>4</sub>	Martin	3	2014-T6	1-3-67	1-25-67	In Test
N <sub>2</sub> O <sub>4</sub>	Martin	1	6A1-4V	1-3-67	1-13-67	10
N <sub>2</sub> O <sub>4</sub>	Martin	1	6A1-4V	1-3-67	2-7-67	34
N <sub>2</sub> O <sub>4</sub>	Martin	1	6A1-4V	1-3-67	2-8-67	35
N <sub>2</sub> O <sub>4</sub>	GD/C	1	5A1-2.55N	1-3-67	1-17-67	14
N <sub>2</sub> O <sub>4</sub>	GD/C	1	5A102.55N	1-3-67	1-19-67	16
N <sub>2</sub> O <sub>4</sub>	GD/C	2	6061-T6	1-3-67	In Test	
N <sub>2</sub> O <sub>4</sub>	Martin	1	7039-T6	1-3-67	7-11-68	555
N <sub>2</sub> O <sub>4</sub>	Martin	1	7039-T6	1-3-67	In Test	
N <sub>2</sub> O <sub>4</sub>	GD/C	1	AM350	1-3-67	10-24-67	294
N <sub>2</sub> O <sub>4</sub>	GD/C	1	AM350	1-3-67	10-25-67	295
C1F5	Martin	1	2014-T6	1-3-67	3-3-67	58
C1F5	GD/C	1	2014-T6	1-3-67	In Test	
C1F5	GD/C	1	AM350	1-3-67	10-24-67	294
C1F5	GD/C	1	AM350	1-3-67	10-25-67	295
C1F5	GD/C	1	6061-T6	1-3-67	In Test	
C1F5	Martin	1	7039-T6	1-3-67	In Test	

TABLE IV. PHASE IV - SUMMARY OF RESULTS

Propellant	Tank	Material	Test Initiated	Test Terminated	Days	Result
N <sub>2</sub> O <sub>4</sub>	Agena	6061-T6	6-12-67	9-19-67	100	No Leak
N <sub>2</sub> O <sub>4</sub>	ULPR	2219-T81	5-21-68			In Test
N <sub>2</sub> O <sub>4</sub>	ULPR	2219-T81	5-21-68			In Test
N <sub>2</sub> O <sub>4</sub>	Bullpup	2014	6-10-68			In Test
N <sub>2</sub> O <sub>4</sub>	Bullpup	2014	6-10-68			In Test
N <sub>2</sub> O <sub>4</sub>	Bullpup	2014	6-10-68			In Test

TABLE V. PHASE V - SUMMARY OF RESULTS

Propellant	Number	Press System	Exp System	Test Initiated	Test Terminated	Days
MHF-5	2	LGG	R.D.	6-9-67		In Test
MHF-5	2	SGG	R.D.	6-9-67		In Test
MHF-5	1	H	R.D.	6-9-67		In Test
MHF-5	2	LGG	S.T.	6-9-67		In Test
MHF-5	2	SGG	S.T.	6-9-67		In Test
MHF-5	2	H	S.T.	6-9-67		In Test
N2O4	2	LGG	R.D.	5-22-67		In Test
N2O4	2	SGG	R.D.	5-22-67		In Test
N2O4	2	H	S.T.	5-22-67		In Test
CIF5	1	SGG	R.D.	6-20-67	10-7-67	20
CIF5	1	H	S.T.	8-4-67	10-23-67	80
CIF5	1	H	S.T.	8-4-67		In Test
*N2O4	2	LGG	R.D.	5-10-67		In Test
N2O4	1	SGG	R.D.	5-10-67		In Test
N2O4	1	H	S.T.	5-10-67		In Test
N2O4	1	H	R.D.	5-10-67		In Test

\*SPEC N<sub>2</sub>O<sub>4</sub>

LGG Liquid Gas Generator  
 SGG Solid Gas Generator  
 H Stored Helium  
 ST Surface Tension  
 RD Rolling Diaphragm

## REFERENCES

1. CR-45-145 - "Improved Leak Detection, Correlation of Actual Leakage with Instrument Indications, Effect of Humidity of Leaks and Categorization of Leak Information." Final Report DSRS 10411, Contract AF04(611)-576, C. Fatino et al, Martin Company, 16 June 1964
2. Midwest Research Institute Interoffice Communication, dated 17 May 1968, subject: "Tracture Analysis on Ti Alloy Weldments", Gordon Gross

**APPENDIX I**

**AFML REPORT MAA 67-5**

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AERONAUTICAL SYSTEMS DIVISION  
AIR FORCE MATERIALS LABORATORY  
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

Materials Application Division

EVALUATION REPORT

INVESTIGATION OF LEAKING WELDMENTS IN 2014 ALUMINUM ALLOY  
 $N_2O_4$  CONTAINERS

REPORT NR: MAA 67-5

DATE: 1 February 1967

PROJECT NR: AFPL 305805223

TYPE EVALUATION: Failure Analysis

MANUFACTURER:

SPEC NR:

SUBMITTED BY: AFSC(AFPL/RPRPT)

ITEM SERIAL NR:

Attn: Lt Goodwin  
Edwards Calif 93523

I. PURPOSE:

Ascertain causes of leaks in nitrogen tetroxide containers of welded 2014 aluminum alloy construction.

II. FACTUAL DATA:

1. Three aluminum alloy cylindrical containers approximately 3-1/8 inches in diameter by 6 $\frac{1}{2}$  inches in length, which had been removed from service in an  $N_2O_4$  system because of leakage, were submitted by AFPL letter dated 18 November 1966 to MAAS for failure analysis. Locations of observed leaks were marked as indicated in Figs. 1 through 4. The tank material was indicated to be 2014 aluminum alloy, joined by heliarc welds with 4043 alloy filler metal.

2. Visual examination of the outer surfaces revealed the presence of general corrosive attack in the weld areas, with selective and severe attack in the weld heat affected zones adjacent to the weld beads. In varying degree, the conditions prevailed on all three containers. Radiography of the weld areas indicated the presence of porosity and variation of weld penetration particularly in the areas in which leaks were observed. It was noted that leakage was very prevalent in the weld overlap areas and at an area which apparently contain a repair weld, see Fig. 2.

3. To determine whether corrosion was a causative factor in the development of the leakage channels, the containers were sectioned at suspect areas to permit examination of the inner surfaces of the containers and to provide metallurgical sections through the welds. There were no visual indications of corrosion on the inner surfaces. Weld penetration of the joints in the .063 inch wall sections was satisfactory as was the soundness of the joints except for weld overlap areas. The circumferential welds joining the .063 inch walls to the .200 inch heads were not found, see Fig. 5., which shows the fractured surfaces of a portion of such

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a weld after forcing it apart. The configuration of the mating surfaces is at variance with the drawings supplied under CD contract AFC4(311)-1C9CC in that the edges of the head plates were not bevelled. Without the bevel it is impractical to attempt to protect the metal surfaces on the back of the joint by a stream of inert gas during welding. Failure to provide adequate protection is confirmed by the lack of fusion and oxidized surfaces of the ball of deposited metal which appears in Fig. 6. Fig. 6 is a view of the inner surfaces in the weld repair area between wall section and head of tank NA-2, the outer surfaces of which are shown in Fig. 2. The result is an open crack capable of causing stress concentration variously estimated at 8 to 10 times that which would be anticipated had a generous fillet been provided.

4. The combination of high stress and surface contamination resulted in crack growth by stress corrosion as indicated by Fig. 7.

### III. CONCLUSIONS:

1. The design of the joint between cylindrical wall and the disc head provides built-in stress concentrations and makes the attainment of a satisfactory weld very difficult.

2. Weld deposits were unsound and penetration was erratic.

3. Leakage initiated at weld flaws.

4. The heat of welding apparently cause metallurgical changes in the base metal which made the heat affected zones particularly susceptible to corrosion by escaping N<sub>2</sub>O<sub>4</sub> after mixing with the outside atmosphere. Repeated application of welding heat, as at weld overlaps or repairs, accentuated the unfavorable condition.

### IV. RECOMMENDATIONS:

1. Modification of welding procedures and container joint design to assure sound welds, with fillets at interior angle joints, are essential to reduce localized stress concentrations and improve the integrity of the containers.

2. Selection of a more weldable aluminum alloy, with attendant increase in weight, is suggested as a means of alleviating the tendency toward selective corrosion at the heat affected zones adjacent to weldments.

**COORDINATION:**

Bennie Cohen  
BENNIE COHEN, MAAS

**PREPARED BY:**

George M. Yoder  
GEORGE M. YODER, MAAS

**PUBLICATION REVIEW**

This report has been reviewed and approved.

W. P. Conrardy  
W. P. CONRADY, Chief  
Systems Support Branch  
Materials Applications Division  
Air Force Materials Laboratory

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Edwards, Calif 93523

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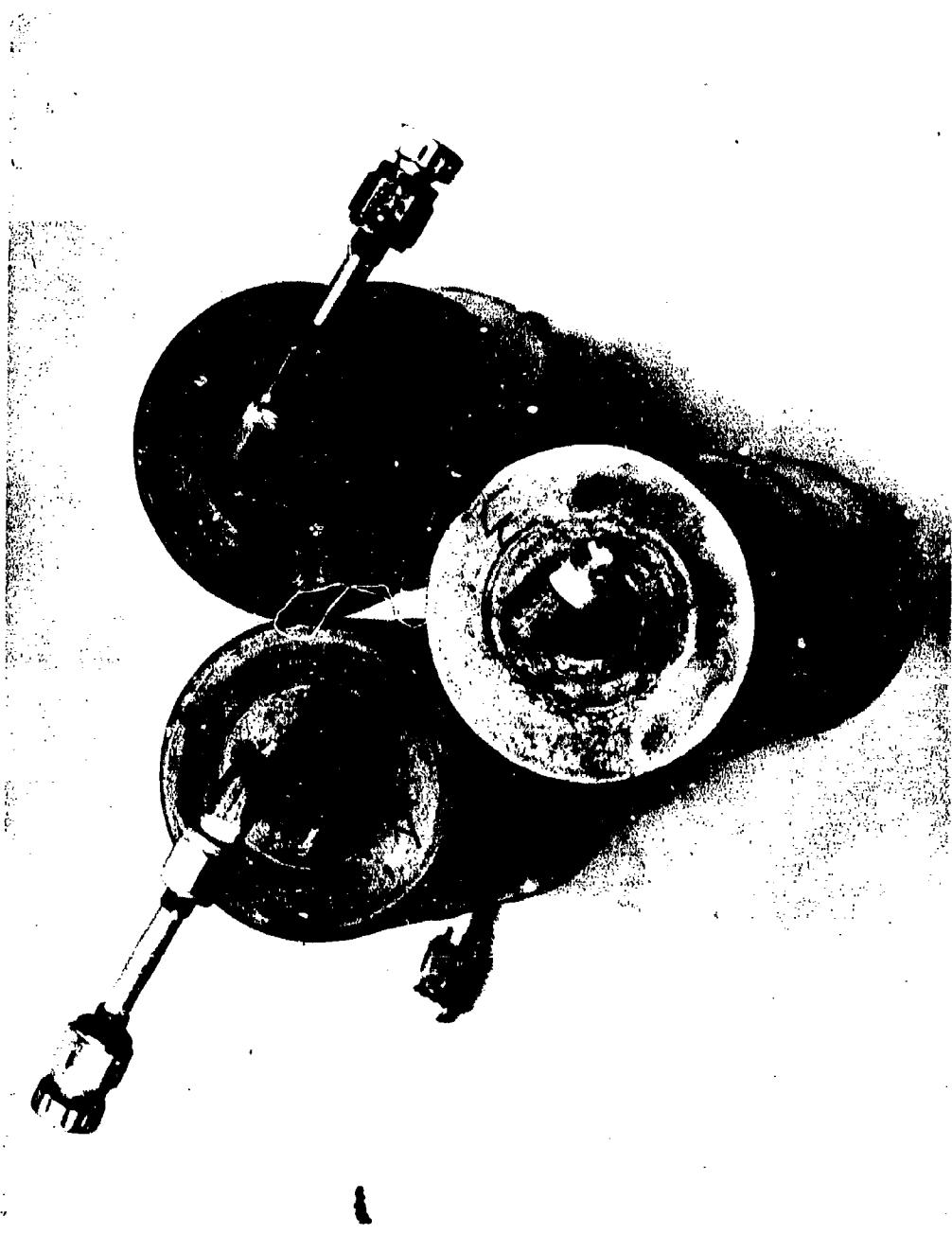
RTD(KTHM/Lt Col Hollyfield)

Appendix I, Figure 1. Observed Leak in 2014 Aluminum Container



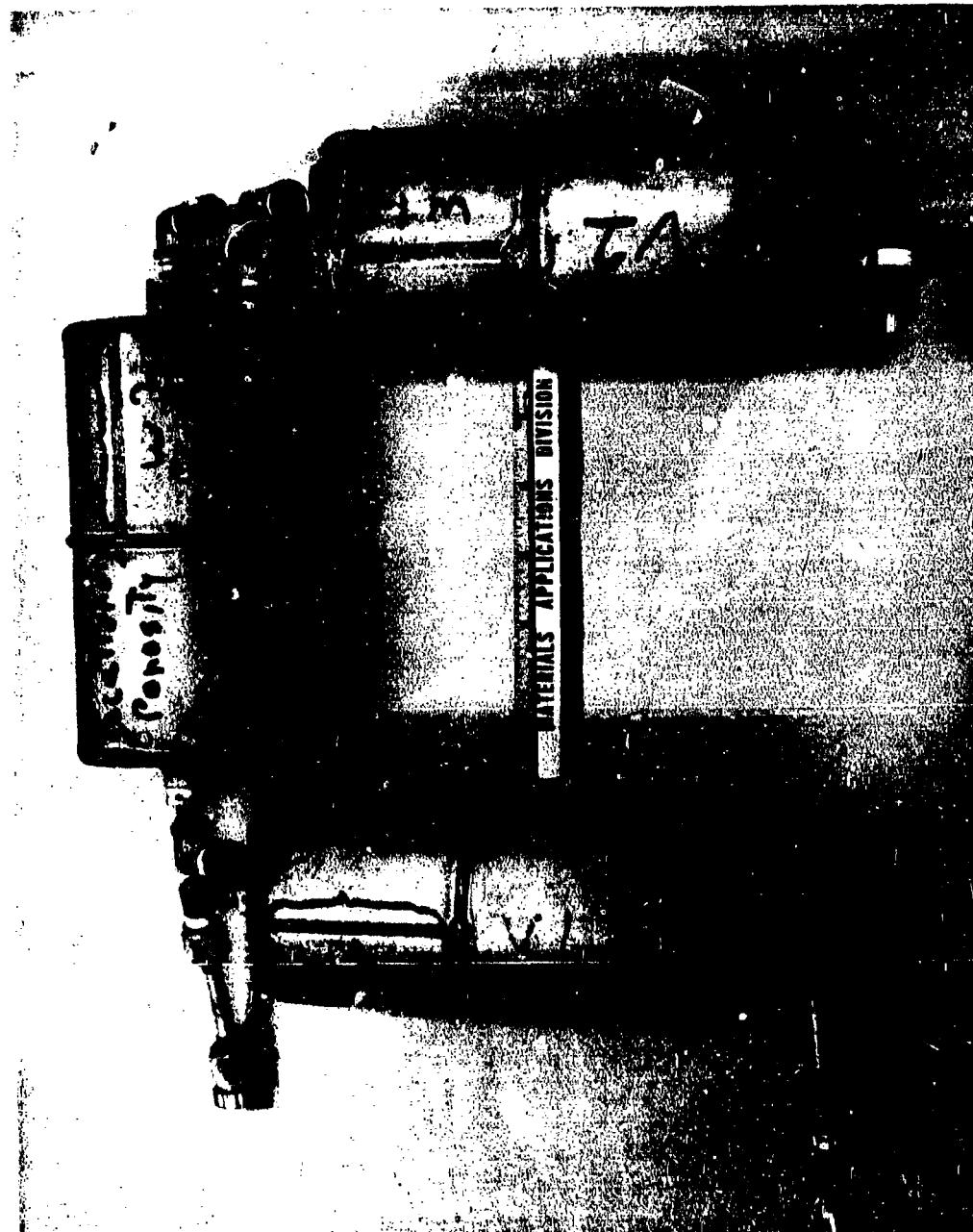
Appendix I, Figure 2. Observed Leak in 2014 Aluminum Container





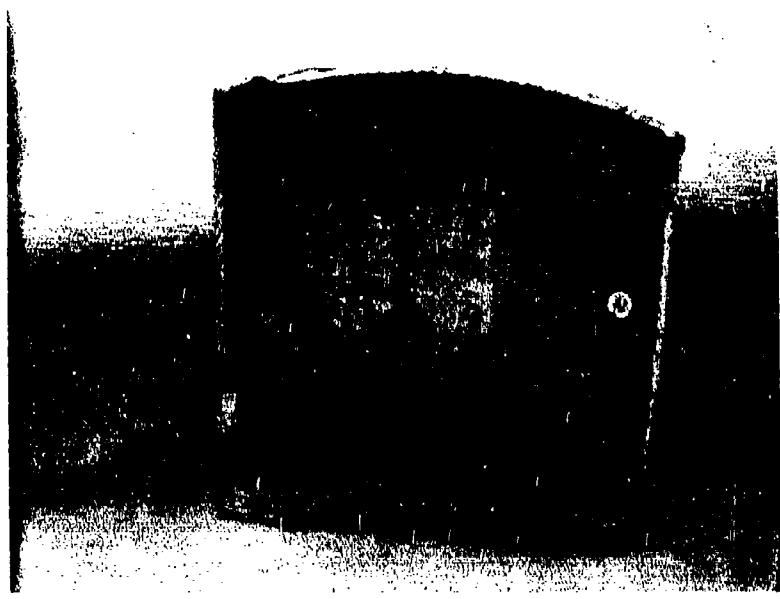
Appendix I, Figure 3. Observed Leaks in 2014 Aluminum Containers

Appendix I, Figure 4. Observed Leaks in 2014 Aluminum Containers





Appendix I, Figure 5. Fractured Surface of Weld  
38



Appendix I, Figure 6. Inner Surface of Weld Repair



Appendix I, Figure 7. Stress Corrosion Crack

# Defense Metals Information Center

Battelle Memorial Institute  
505 KING AVENUE  
COLUMBUS, OHIO 43201  
AREA CODE 614. TELEPHONE 299-3181

January 29, 1968

Mr. J. E. Branigan  
Code RPRPT  
Air Force Rocket Propulsion Laboratory  
Edwards, California 93523

Dear John:

We have completed our examination of the two 15 gallon tanks which failed in the long term storage tests with red  $N_2O_4$ . One tank was constructed of Ti-6Al-4V, the other of 2014 T-6 aluminum. Our finding may be summarized as follows:

## Titanium (Ti-6Al-4V) Tank

It is our understanding that the 15 gallon tank for the long term storage test of  $N_2O_4$  was manufactured by Martin-Denver. After cleaning and decontamination according to Martin Company process specification EPS 50063, it was recleaned by EAFB as follows:

1. Solvent decrease - Perchloroethylene
2. Rinse - Alcohol and deionized water
3. Dry -  $N_2$  gas

After leak testing at 150 psig with a mixture of  $N_2$  and He the tank was filled with red  $N_2O_4$  at 100 psig and stored in an environmental chamber at 85 F and 85 percent relative humidity. Leaking  $N_2O_4$  was detected visually after about 35 days on test. The tank was emptied and retested for the leak points.

Two areas on the tank were marked where leaks were detected. The most obvious of these was a crack adjacent to a weld bead where the weld bead overlapped. The second area was on the side wall of the tank and not associated with a weld. No obvious leak point could be seen on this region.

Several metallographic sections were taken from this second area and carefully examined. No cracks or flaws which could account for the leak were found. In addition, no evidence of any attack was noted on the surface prior to sectioning. The absence of any detectable flaw by the section-and-search technique suggests that the leak probably was the result of helium migrating over the surface of the tank from the large crack at the weld.

January 29, 1968

The tank was sectioned at the weld as shown in Figure 1 and the crack examined metallographically. Figure 2 contains a photomicrograph of the large crack adjacent to the weld bead and four smaller cracks toward the edge of the heat affected zone. Figure 3 shows a higher magnification of the small cracks in Figure 2. Figure 4 is a more detailed view of the large crack. All of the cracks propagated from the inside of the tank outward.

Hardness measurements were made at the points marked on Figure 2 and the results of these measurements are listed directly below the marks on the photo (KHN). Contamination of the weld would have been expected to result in much higher hardnesses and a much greater range of hardnesses than was observed. In view of the structure and hardness the possibility of weld contamination does not appear to have been a significant factor in the failure.

The morphology of the cracks observed is typical of that normally associated with  $N_2O_4$  stress corrosion cracking of the titanium-6Al-4V alloy. There seems little doubt that the failure of the tank was anything other than stress corrosion cracking. While little or no differences in susceptibility to stress corrosion cracking in  $N_2O_4$  has been observed for welds versus parent material, the fact that the cracking in this case is associated with the weld is not expected. For example, pressurizing the tank to 100 psig results in a hoop stress of only 13.3 KSI. This is far below the threshold stress of about 40 KSI which has been found to initiate cracking of titanium in  $N_2O_4$ . It is interesting to note that the crack occurred at a weld overlap or severed area. Such a condition would be expected to give rise to highly localized stresses which coupled with the applied load of 13.3 KSI are sufficient magnitude to initiate and propagate a stress corrosion crack. The fact that four other titanium tanks (one Ti-6Al-4V, three Ti-5Al-2.5Sn) have failed in your  $N_2O_4$  storage studies at a double pass weld, to junction weld and/or weld repair is further evidence that such conditions endure high stresses necessary for cracking in  $N_2O_4$ . It is doubtful that any failures would have occurred at least in the short exposure time if the welds had been of a different configuration.

#### Aluminum (2014-T6) Tank

The same procedural details listed for the titanium tank (1st paragraph of the previous section) apply to the 15 gallon aluminum tank, with the exception of the Martin Company cleaning procedure and the time failure. The aluminum tank failure was detected after 22 days in test. As before, failure was determined by visual observation of the leaking gas. Extensive chemical attack has occurred in the region of the largest of two



2x

C-2746

Appendix II, Figure 1. Double-Pass Weld With Adjacent Crack Viewed From Inside of Tank



70X    323    330    345    347    354    354    C-2747m2748

Appendix II, Figure 2. Heat-Affected Zone of the Double Weld Showing the Two Areas Where Cracks Were Detected



200X

C-2749

Appendix II. Figure 3. Small Cracks Toward the End of the Heat-Affected Zone



100X

C-2750

Appendix II, Figure 4. Large Crack Adjacent to Weld Bead

Mr. J. E. Branigan

7

January 29, 1968

leaks on the outside of the tank. This attack took place primarily along the weld heat affected zone, after the tank leaked. In this case,  $N_2O_4$  reacted with water from the high humidity environment forming nitric acid which rapidly attacked the aluminum alloy. This is illustrated in Figure 5. The severity and extent of the external corrosion surrounding this  $N_2O_4$  leak suggests that some time had elapsed between the initial leak and the actual observation of the leak. It is pointed out that the attack occurred after the leak and was not the cause of the leak.

The major leak point at the tee junction of the weld was sectioned. Numerous large gas voids with an interdendritic crack network running from hole to hole was found. This is shown in Figure 6. There is no doubt that the weld itself played a major role in the failure at this point. The number and size of the gas voids in the weld is reason enough to conclude that the weld was substantially weakened at this point. A sound weld of good quality would not be expected to suffer corrosion in the  $N_2O_4$ .

A number of small white spots were observed adjacent to the weld in the vicinity of the leak at the tee. Similar spots were also found either in or near the entire area of the top circumferential weld. The center of each spot contains a small fissure oriented perpendicular to the direction of major stress on the tank. This consistency of orientation perpendicular to the major stresses suggests a stress enhanced failure mechanism.

The largest of these white spots (located about 4 inches to the right of the tee in Figure 5) was marked as a second leak point on the outside of the tank.

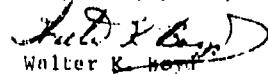
Figure 7 is a cross sectional view of the fissure in the largest of the white spots where the second leak was marked.

This type of intergranular corrosion is a common observation on aluminum alloys. An applied stress enhances such attack and this would explain the unidirectionality observed. The initial breakdown of the normally passive  $N_2O_4$ /aluminum system at these points is believed to have been caused by surface contamination.

Contamination could have been present from many sources such as water condensate during shipping or cleaning fluid residue. The combined action of some contaminant and the  $N_2O_4$  environment is the probably cause of the accelerated attack.

The Defense Metals Information Center was glad to be of assistance in the examination of the tanks. If you have any questions, or if we can be of further assistance please let us know.

Very truly yours,

  
Walter K. Bern  
Chief  
Corrosion Research Division

WKB:rs



2x

C-2751

Appendix II, Figure 5. The Weld Tee Junction of the Aluminum Tank  
Showing the Extensive External Corrosion From  
Nitric Acid

C-2753

6b

C-2752

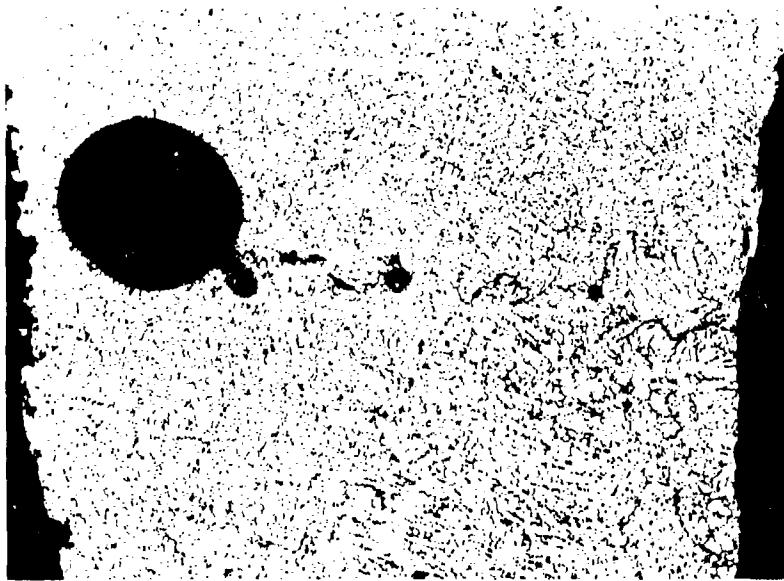
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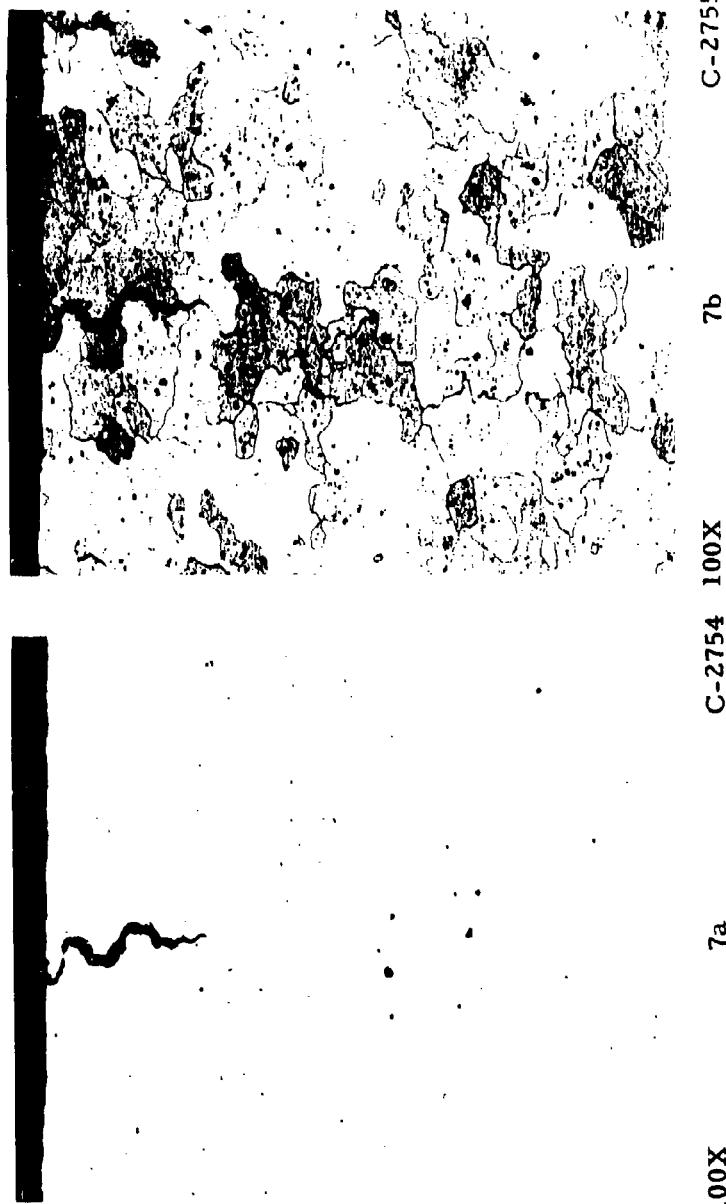
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6a

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Appendix II, Figure 6. The (a) Unetched and (b) Etched Cross Section of the Crack Through the Weld Tee Junction





Appendix E. Figure 7. The (a) Unetched and (b) Etched Cross Section Through the Largest of the White Spots Containing Intergranular Fissures

## Security Classification

## DOCUMENT CONTROL DATA - R &amp; D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY (Corporate author) Air Force Rocket Propulsion Laboratory Edwards, Calif. 93523		2a. REPORT SECURITY CLASSIFICATION Unclassified
		2b. GROUP N/A
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4. DESCRIPTIVE NOTES (Type of report and inclusive dates) Interim Report, January 1967 through March 1969		
5. AUTHOR(S) (First name, middle initial, last name)  John E. Branigan		
6. REPORT DATE April 1969	7a. TOTAL NO. OF PAGES 58	7b. NO. OF REFS 2
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b. PROJECT NO. 3058	9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report)	
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11. SUPPLEMENTARY NOTES	12. SPONSORING MILITARY ACTIVITY  See Block 1	
13. ABSTRACT Air Force weapons systems require long-term maintenance-free storage, preferably under uncontrolled environmental conditions. Liquid propulsion system components must be capable of satisfactory operation after years of highly reactive propellants while retaining the propellant without leakage under severe ambient conditions of temperature and relative humidity. Oxidizer leakage caused by improper component design and severe ambient storage conditions has presented serious operational problems.		
<p>The Air Force Rocket Propulsion Laboratory (AFRPL) has initiated a program to investigate the storability of liquid system components and tankage under extreme conditions of relative humidity and temperature. A variety of system components and tankage materials are being evaluated for long-term storability with storable liquid rocket fuels and oxidizers. Storage conditions are 85°F temperature and 85% relative humidity for oxidizer systems and 70 to 150°F temperature for fuel systems. The propellants under test are N<sub>2</sub>O<sub>4</sub>, C<sub>1</sub>F<sub>3</sub>, C<sub>1</sub>F<sub>5</sub>, and MHF-5. Tankage materials under test are various alloys of aluminum, steel, and titanium.</p> <p>The results of almost 2 years of testing on a representative number of tankage materials have indicated that leakage of propellant can occur as a result of improper weld joint design, inadequate quality control in fabrication and inadequate acceptance leakage testing. Factors which can contribute to the development of oxidizer leakage are a high ambient relative humidity (&gt;30%) and stress corrosion cracking susceptibility of the tank material in combination with the propellant and trace quantities of foreign compounds/elements in the propellant.</p>		

DD FORM 1 NOV 68 1473

~~UNCLASSIFIED~~

Security Classification

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Propellant storage Environmental effects Propellant leakage Corrosion Oxidizer Leakage Stress corrosion Intergranular corrosion Hydrazine blend storage Packaged propulsion system storability Stress corrosion cracking Rocket propellants Leak detection Propellant tank quality control						

Unclassified

Security Classification